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DEVELOPMENT OF NEW DECON GREEN®: A HOW-TO GUIDE FOR THE RAPID DECONTAMINATION OF CARC PAINT

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14. ABSTRACT
This study presents the further refinement of the original Decon Green® "Classic" to the New Decon Green® formula. Four main problems were identified with the "Classic": 1) limited capacity for non-traditional agents; 2) long-term stability; 3) homogeneity; and 4) material compatibility, especially with paints, M40 Mask lenses, and HMMWV light housings. These problems have been solved, but at the expense of decon efficacy of Chem Agents (not Bio agents) for soft/sorptive materials such as Chemical Agent Resistant Coating (CARC) paint. The Bio efficacy of New Decon Green® remains comparable to Decon Green® Classic as Bio agents do not penetrate/soften materials. Moreover, Chem efficacy still remains better than other peroxide-based decontaminants such as DF200, especially for paint-penetrating HD. Finally, a simple model is presented to extrapolate measured contact hazard levels to potential vapor hazard levels. Off-gassing data for HD and GD on CARC paint is also discussed along with the subjective nature of this test, its ambiguous results, and the problem of relating the results to a true, accurate vapor hazard level. Currently, contact hazard and/or total extraction (residual hazard) remain the only unambiguous tests to verify decontamination efficacy on surfaces such as CARC where substantial agent remains following decontamination.

15. SUBJECT TERMS

Decon Green	Decontamination	Paint softening	GD	VX	TVX
Off-gassing	Vapor hazard	THD	CARC	DS2	THD
Anthrax	Contact hazard	TGD	DF200		

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PREFACE

The work described in this report was authorized under Project No. CDEC3007, the U.S. Army Edgewood Chemical Biological Center Tech Base Program. The work was started in September 2002 and completed in February 2005.

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DEVELOPMENT OF NEW DECON GREEN®: A HOW-TO GUIDE FOR THE RAPID DECONTAMINATION OF CARC PAINT

1. INTRODUCTION

In early 2002, the original Decon Green®[*] "Classic" formula resulted from laboratory testing. It proved not only to be superior to DS2 for the decontamination of agents on Chemical Agent Resistant Coating (CARC) paint, but also provided an additional capability for the decontamination of bio agents such as anthrax.[1] Subsequently, Decon Green® Classic underwent scale-up and operational testing, and registration of the "Decon Green" trademark was sought. During the testing period, several problems were uncovered, including 1) limited capacity for non-traditional agents (NTAs); 2) long-term stability; 3) homogeneity; and 4) material compatibility, especially with paints, M40 Mask lenses, and HMMWV light housings. These apparent failings prompted a reformulation effort which ran concurrent to the continued large-scale testing of Decon Green® Classic.

The current report on New Decon Green® details solutions and compromises undertaken in an attempt to solve the above stated problems. Where appropriate, related, and even antagonistic, effects are grouped together for discussion. Chemical and Biological decontamination data are also presented to compare and contrast New Decon Green® with Decon Green® Classic and DF200, another peroxide-based decontaminant urgently adopted by the Army in preparation for the 2003 Iraq War.[2]

2. EXPERIMENTAL PROCEDURES

2.1 Chem Tests

2.1.1 Reactor Test

Reactions were carried out in glass-jacketed reactors fitted with mechanical stirrers at 10, 25, and 50 ºC. Reactions were simultaneously run in triplicate in three identical reactors. In a typical run, 50 mL of decontaminant was added to each of the three reactors, the stirrers were started, and 1 mL of agent was added to each of the three reactors. At desired time points, 59-µL samples were removed from the reactor and quenched with 1 mL of 0.2 M sodium sulfite and 0.2 M sodium carbonate, and extracted with 2-mL chloroform. The chloroform layer was analyzed for residual agent by Gas Chromatography/Atomic Emission Detection (GC/AED).

[*] "Decon Green" is a registered trademark of the Department of the Army.

2.1.2 Panel Test

CRC-painted panels, 2-in. diameter (D) each, were employed. Six replicates were used for each decontaminant. The panels were contaminated with 2-μL drops of agent to yield a contamination density of 10 g/m^2. Droplets were spread around with a piece of parafilm to form a thin, uniform film of the agent on the panel. The panels were covered to prevent excessive evaporation in the fume hood and allowed to stand for 1 hr. A volume of 1-mL decontaminant (1:50 agent to decontaminant ratio) was applied to the panels, evenly distributed with the pipette tip, and allowed to stand covered for 15 min. Excess decontaminant was then poured off and the panels were rinsed with two 20-mL portions of water. The panels were allowed to air dry in a vertical position in the fume hood for 2 min. After drying, the panel was placed on a warming surface set at 30 ºC where contact tests were conducted by placing the following, in order, on top of the panels for 15 min: 2 in. latex disk; 2 in. aluminum foil disk; 2 in. D-1 kg weight with foam padding on the bottom. After removal from the panel, the latex and aluminum foil disks were extracted in 20-mL chloroform (containing 1-mL/L thiolane to quench any remaining peroxide) for 1 hr. After 30 min, another contact test was conducted in the same manner. After the second contact test, the panel itself was extracted in 20-mL chloroform (containing 1-mL/L thiolane) for 1 hr to determine the residual amount of agent remaining on the panel. Solutions were analyzed by Gas Chromatography/Flame-Ionization Detector (GC/FID) to determine the amounts of agent recovered.

2.1.3 Paint Softening Tests

After the treatment of the CARC-painted panels by agents and/or decon solutions, the hardness of the CARC paint was tested by using a pencil hardness gage (Paul N. Gardner Co., Inc.).

2.1.3.1 Softening by Agents

Two-inch diameter CARC-painted aluminum panels were placed, painted-side down, into 1.4 mL of agent in a Petri dish. The Petri dish was placed inside a sealed weighing dish to prevent evaporation of the agent. For hardness testing, the panels were removed from the agent, blotted dry with a Kim-wipe, and allowed to air dry further before testing.

2.1.3.2 Softening by Decontaminants

The 2 in. CARC-painted panels were subjected to five successive 15-min applications of both Decon Green® Classic and New Decon Green® (see Section 2.1.2). After each application of decontaminant, the panel was rinsed with water and allowed to dry overnight prior to hardness testing.

2.1.4 Test of CARC Panels to MIL-C-53039A(ME)

CARC-painted aluminum panels were tested for their chemical agent resistance as described in MIL-C-53039A(ME), "Military Specification, Coating, Aliphatic Polyurethane, Single Component, Chemical Agent Resistant" with one modification: 20-cm^2 coupons were

contaminated rather than the prescribed 5-cm^2 area on a larger panel. The results were then scaled to the requisite 5-cm^2 area. Two-inch diameter (20 cm^2) panels were unwrapped and exposed to ambient room air for 4 days before being placed into a 105 ºC oven for 3 days. The panels were allowed to cool to room temperature. To carry out the test, six panels were placed on aluminum foil and each was contaminated with 10-HD drops of approximately 2-µL volume. The drops were spread with a piece of parafilm to completely wet the surface of the panels. The panels were covered with inverted Petri dishes (to prevent evaporation of the HD) and allowed to stand for 30 min. The contaminated surfaces of the panels were then rinsed five times with isopropanol (IPA) and the back sides were rinsed twice with IPA. The IPA was allowed to evaporate from the panels, which took about 1 min. The panels were sealed in vapor cups and vapor collection was initiated at 50-cm^2/min airflow using bubblers containing 10-mL diethylphthalate (DEP). Vapor was collected for a 24-hr period. The DEP from the bubblers was transferred to glass scintillation vials. A 100-µL aliquot of the DEP was diluted with 900-µL chloroform (1:10 dilution) in a Gas Chromatography (GC) vial prior to GC/FID analysis to determine the amount of recovered HD.

2.2 Bio Tests

2.2.1 Spore Preparation

All spores were prepared according to standard microbiological practices, as outlined by Leighton and Doi.[3]

2.2.2 Suspension Tests with *Bacillus atrophaeus* Spores

All testing was conducted in triplicate. Suspension tests were conducted by suspending 1 x 10^9 *B. atrophaeus* colony forming units per milliliter (CFU mL^{-1}) in sterile water. The suspension was thoroughly mixed by vortexing. Then 10 µL of the spore suspension were dispensed into 9-microcentrifuge tubes. Three hundred ninety microliters of decontaminant or phosphate buffered saline (PBS) were dispensed into the microcentrifuge tubes. The decontaminants were used within 1 hr of preparation. In addition to the positive (PBS) controls, each test included a PBS negative control that did not contain spores. The negative control was handled in the same manner as the other test samples. After addition of the decontaminant or PBS and vortexing, each sample was then mixed thoroughly by vortexing and placed on the thermomixer at 20 °C for 15 min. At the end of 15 min, 600 µL of 30% sodium metabisulfite was added to each tube to neutralize the decontaminant. The samples were centrifuged at 20,800 x g for 5 min, the supernatant was removed, and the pellets were resuspended in 1-mL PBS a total of two times before resuspending each sample in PBS. The positive control samples were resuspended in a final volume of 1 mL. The positive controls were serially diluted in PBS, after which 100 µL from each dilution tube was spread on trypticase soy agar (TSA) plates in triplicate. Based on previous test results (data not shown) all other samples were resuspended in 120-µL PBS. For each sample, the entire 120 µL was spread on a single TSA plate. All TSA plates were incubated overnight at 37 °C. The CFUs were counted the next day as an indication of the spore viability.

2.2.3 Three Step Method Test with *B. anthracis* Spores

For testing on specific materials (CARC paint and rubber), the Three Step Method was employed, based on the ECBC Standard Operating Procedure for the Three Step Method.[4,5]

Briefly, 0.5 cm^2 materials were autoclaved and inoculated with ~10^6 CFU and permitted to dry at room temperature. Each contaminated material was then placed in a microcentrifuge tube. Four hundred microliters of each decontaminant or water (control) were added to the microcentrifuge tubes. The decontaminants were used within 1 hr of preparation. The samples were permitted to sit at 23 °C for 30 min. At the end of 30 min, 600 µL of ice cold Luria Bertani Broth (LB broth) or 30% sodium metabisulfite were added to each tube. This sample was now labeled Fraction A. The material was removed from each tube and placed in another tube containing 400 µL of sterile, room temperature water (Fraction B).

Fraction A was centrifuged and then washed two times by centrifugation (13,000 rpm for 6 min) and resuspended in ice cold LB broth. Fraction B was sonicated for 5 min at room temperature before adding 600 µL of ice cold LB broth, vortexing, and transferring the materials to tubes labeled Fraction C. Fraction C microcentrifuge tubes contained 400 µL room temperature LB broth. Fraction B tubes were centrifuged one time at 13,000 rpm for 6 min before resuspending in ice cold LB broth. Fraction C tubes, still containing the materials, were incubated at 37 °C for 30 min before adding 600 µL of ice cold LB broth to each tube. Control sample Fractions A and B were resuspended in a final volume of 1 mL of ice cold LB Broth, then serially diluted in LB broth before plating on TSA plates. Test sample Fractions A and B were resuspended in a final volume of 120 µL, so that the entire sample could be plated out onto TSA plates. One hundred microliters were plated from all Fraction C tubes. All the plates were incubated overnight at 37 °C before the CFUs were counted as an indication of spore viability.

2.2.4 Glass Slide Surface and Suspension Decon of *B. anthracis* Spores and *Yersinia pestis* Cells

For the glass slides, 100-µL aliquots of 1 x 10^8 spores or 1 x 10^7 cells were deposited onto sterile microscope slides. The slides were dried in a BioSafety Cabinet for at least 4 hr. A total of five slides were used per experiment (2 control and 3 experimental). After the slides dried, 0.5 mL sterile H_2O or freshly prepared decon solution was added via a pipette. The pipette tip was used to scrape/scrub the surface to gently dislodge the spores/cells. The water or decon solution was recovered and placed in sterile 1 mL eppendorf tubes. A second 0.5 mL portion of water or decon solution was added to the slide. Both the recovered solutions and slides were allowed to stand for 15 min. After 15 min the solutions were serially diluted to 10^{-6}. The slides were placed in 50 mL conical tubes with 10 mL sterile H_2O. The slides were vortexed for 1 min to dislodge any remaining spores/cells. The wash from the slides was serially diluted to 10^{-4}. Volumes of 100 µL of the dilutions were spread plated in triplicate on TSA plates, including the original tubes, and incubated at 37 ºC overnight. The next day, the plates were enumerated to calculate the CFU survivors.

For suspensions, 10 µL of 1 x 10^8 spores or 1 x 10^7 cells were added to a sterile 1 mL eppendorf tube. Five tubes were prepared (2 controls and 3 experimental). Either 990 µL sterile H_2O or freshly prepared decon solution was added. The tubes were placed on a shaker for 1 hr and then centrifuged at 14,000 rpm for 10 min. The supernatant was removed, 1 mL sterile H_2O was added, and the tube was vortexed for 10 s. The solutions were serially diluted in sterile H_2O to 1 x 10^{-5}. Volumes of 100 µL of the dilutions, including the original tube, were spread plated on TSA plates and incubated at 37 ºC overnight. The next day the plates were enumerated to calculate the CFU survivors.

3. RESULTS AND DISCUSSION

3.1 Solution Homogeneity and Material Compatibility

The main solvent in Decon Green Classic®, propylene carbonate, is not miscible with water; thus, it tends to phase separate at amounts greater than about 10 vol%. Previous studies conducted at the Naval Surface Warfare Center (NSWC) examining the effect of candidate Decon Green® formulas containing various levels of propylene carbonate found a noted decrease in CARC paint softening at levels of 10 vol% or less.[6] Merely decreasing the amount of propylene carbonate from 55 vol% (Decon Green® Classic formula) to 10 vol% in the New Decon Green® formula simultaneously provided for both a homogeneous solution and an anticipated, improved material compatibility.

To replace the 45 vol% of propylene carbonate removed from the formula, 20 vol% propylene glycol and an additional 25 vol% of catalyst solution were added. Rather than just making up the balance with water, propylene glycol was selected for four reasons: 1) to retard drying; 2) to maintain a low freezing point; 3) to maintain good surface adherence; and 4) because it is edible.[*]

Additional testing by NSWC confirmed the improvement in material effects for New Decon Green®: 1) CARC paint softening test passed (zero reduction in hardness, even after 24-hr immersion) and 2) M40 Mask Lens hazing test passed (only 35.23% haze change after 24 hr immersion).[9] By comparison, it should be noted that Decon Green® Classic resulted in softening of CARC paint by 4-hardness classes (less than 2-hardness classes is the passing criteria) and caused an approximate 806.53% haze change in the M40 Mask Lens (less than 500% change is the passing criteria).

An additional material found to suffer adverse effects during operational testing[10] is the polycarbonate HMMWV light housing, which had been observed to crack apart following exposure to Decon Green® Classic during actual spray testing on vehicles. Work at NSWC confirmed this weakening of the housing, finding that exposure to Decon Green® Classic causes both a 12.5% hardness change and a 0.81% weight loss (sorption change).[9] Furthermore, the housing became very brittle, to the extent that it could easily be separated at the seams with only

[*] While neither fat nor protein, the body perhaps best recognizes propylene glycol as "some kind of carbohydrate" since metabolism apparently occurs by the usual pathways (to acetate, lactate or glycogen).[7] Propylene glycol affords a nutritional value of 570 kcals per 100 g.[8]

slight force. Testing by NSWC of the New Decon Green® with the housing found only minimal change in hardness (3.33%) and weight change (sorption, -0.04%).[7] Additionally, no resulting brittleness of the light housing was noted. It is thus reasonably anticipated that HMMWV light housings will be unaffected under future spray testing with New Decon Green®, but this remains to be seen.

Although great strides were achieved in material compatibility, unfortunately, these gains came at a great cost to decon efficacy on CARC painted surfaces (see below).

3.2 Long-Term Stability and NTA Efficacy

In a now infamous and widely, but improperly, publicized event, Decon Green® Classic was accidentally discovered to undergo latent, but spontaneous heating with concomitant vigorous foaming during an initial large scale (100 gal) mixing and spraying test in September 2002.[*] This exhilarating occurrence does not happen immediately upon mixing; rather, it takes a few hours to develop. So a 2-hr use limitation was subsequently placed on mixed Decon Green® Classic. Although mixing-on-the-fly applicators, such as the Intelagard® DG-Specific Falcon™ (see below), effectively gets around this long-term stability problem (as decon is only mixed as it is sprayed), it was still considered expedient to formulate the decontaminant so as not to have such a restriction on its use.

Looking into the long-term stability issue, it was found that the high pH provided by the potassium carbonate (K_2CO_3) activator/buffer of Decon Green® Classic primarily contributed to the problem, with an additional exacerbation inflicted by the potassium molybdate (K_2MoO_4) activator. However, high pH also allows for fast decontamination of NTAs and VX. Thus suitable, alternative buffers/activators were needed to afford a compromise between long-term stability and NTA/VX reactivity.

In keeping with the principle of Decon Green® development, the buffer candidate(s) needed to be environmentally-friendly, and even edible if possible. Edible potassium bicarbonate ($KHCO_3$), which had been examined in early development work on Decon Green® Classic but discarded in favor of the higher pH, edible K_2CO_3 for better VX/NTA reactivity, was found to afford greater long-term stability. Additionally, edible potassium citrate was found to afford exceptional, even greater long-term stability. By using a mixture of these two food additives and retaining the edible, vitamin ingredient molybdate (albeit in lower concentration), an apparent indefinite long-term stability could be achieved. However, to gain acceptable VX/NTA reactivity, large amounts of potassium bicarbonate and potassium citrate were required. This necessitated raising the amount of water-catalyst solution in the New Decon Green® formula to 30 vol%.

Although $KHCO_3$ provides for long-term stability, unlike K_2CO_3, it tends to precipitate out of solution on cooling. Thus a low temperature limitation of 5 ºC (41 ºF) exists for cold weather use of New Decon Green®. In other words, the low temperature utility of

[*] Wagner, G.W.; Procell, L.R.; Sorrick, D.C. U.S. Army Edgewood Chemical Biological Center; unpublished results.

Decon Green® Classic (–25 ºC/–13 ºF) has been lost in developing the new formula. However, a "Cold and Artic Weather Type" (CA^2WT) Decon Green®, which does not freeze or precipitate at –15 ºC, has been developed, although its long-term stability is slightly less than that of New Decon Green® (see below).

3.3 Toxicity/Environmental-Acceptability of Ingredients

As mentioned above, throughout the development of Decon Green® only environmentally-friendly and, where possible, edible ingredients are employed; thereby, virtually guaranteeing that the decontaminant will not be toxic to personnel (when properly used) and will not present a persistent hazard to the environment. In Table 1, the various ingredients in both New Decon Green® and Decon Green® Classic are shown grouped according their degree of benignity, i.e., food-additive; approved for oral use; approved for topical use; and approved for use in the environment. Propylene glycol even provides nutritional value.[8] It is noteworthy that the only ingredient not approved for internal or external use is the non-ionic surfactant, Triton® X-100, a widely used dispersant for spraying pesticides.[*] Photographs of everyday products containing these exact ingredients, or similar ingredients in the case of the non-ionic surfactant, are shown in Table 2.

The hydrogen peroxide (VHP) component used in both New Decon Green® and Decon Green® Classic requires additional comment. While VHP in dilute amounts, i.e., 3% or less, is not only not harmful, but beneficial when applied to cuts, abrasions, and even for oral health (1.5%), it becomes problematic at higher concentrations (e.g., at above 10%, irritation and blistering of skin occurs). The high concentration of VHP (35%) was selected for use in New Decon Green® and Decon Green® Classic for three main reasons: 1) 35% is a common, inexpensive industrial grade with a freezing point of –33 ºC, so it may be used in cold weather; 2) higher concentrations of VHP afford increased decon efficacy (both chem and bio); and 3) 35% VHP, in limited quantities, is permissible for flight on commercial aircraft. Moreover, larger amounts are transportable on military aircraft, up to the non-bulk limit (119 gal/882 lb), provided proper packaging is employed.[11]

It is primarily because of this high concentration of VHP that proper protective clothing is required for the use and application of New Decon Green® and Decon Green® Classic. It is fortunate, however, that VHP decomposes and dissipates quickly on contact with the environment, forming only water and oxygen. Thus, concentrated VHP on the ground is not long-lived, so it is not a persistent hazard to personnel. Indeed, it is worth stating again that dilute VHP is not hazardous to personnel. It should be noted that the situation is quite different for chlorine-based decontaminants: 1) there is no approved internal or external use for chlorine and 2) once in contact with organic matter chlorine forms toxic dioxins, thereby creating a persistent environmental hazard. Thus, chlorine-based decontaminants are not nearly as environmentally, or personnel-friendly, as VHP.

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[*] Specimen label for Remedy®; Dow AgroSciences LLC: Indianapolis, IN.

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Table 1. New Decon Green® and/or Decon Green® Classic Ingredients

Food-Additive	Oral Use	Topical Use	Agricultural Use
Propylene Glycol	VHP, 1.5%	VHP, 3%	Triton® X-100
Potassium Citrate		Propylene Carbonate	
Potassium Bicarbonate			
Potassium Carbonate			
Potassium Molybdate			

Table 2. Consumer Products Containing Identical/Similar New Decon Green® and Decon Green® Classic Ingredients

Propylene Glycol		
Cold Remedy	Condiment[a]	Makeup
		

[a] Contains propylene glycol alginate. The propylene glycol moiety is absorbed[7] during digestion.

Table 2. Consumer Products Containing Identical/Similar New Decon Green® and Decon Green® Classic Ingredients (Continued)

<table>
<tr><th colspan="3">Propylene Carbonate</th></tr>
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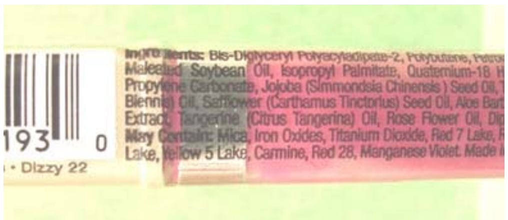
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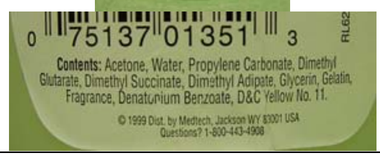
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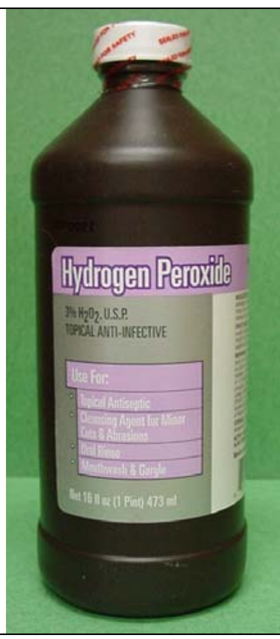
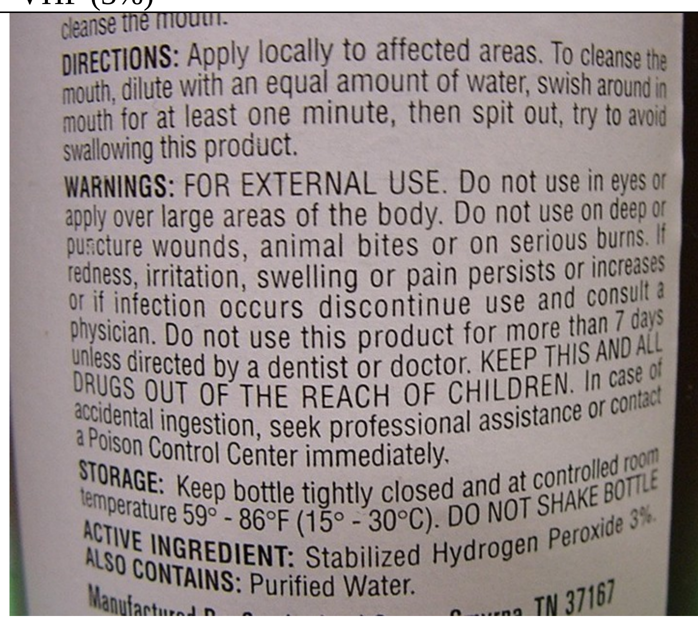
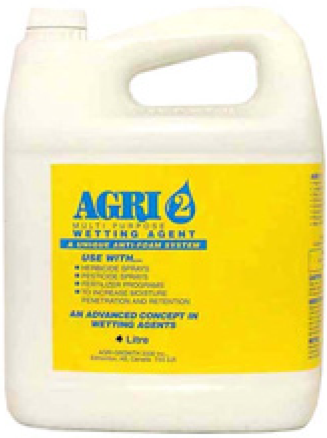
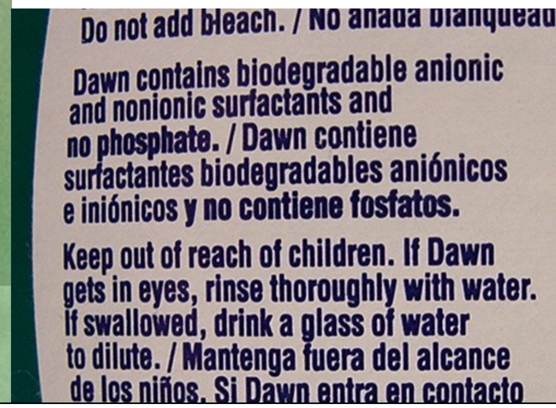
<table>
<tr><th colspan="2">VHP (3%)[a]</th></tr>
<tr>
<td>
</td>
<td>
</td>
</tr>
<tr><td colspan="2">[a] 3% approved for dermal contact; 1.5% approved for oral rinsing.</td></tr>
</table>

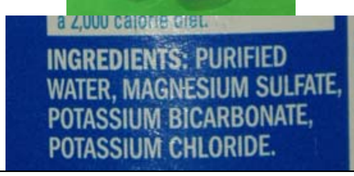
Table 2. Consumer Products Containing Identical/Similar New Decon Green® and Decon Green® Classic Ingredients (Continued)

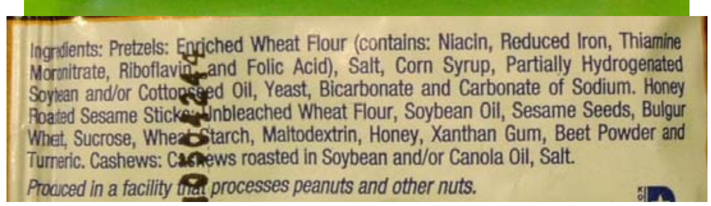
Non-ionic Surfactants





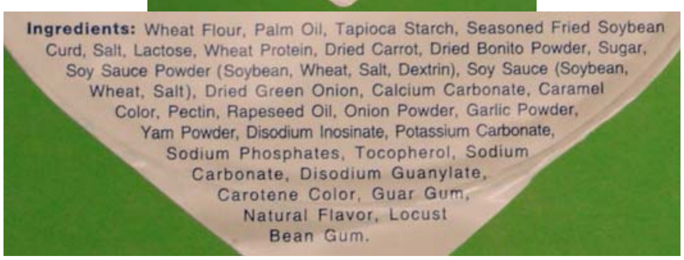
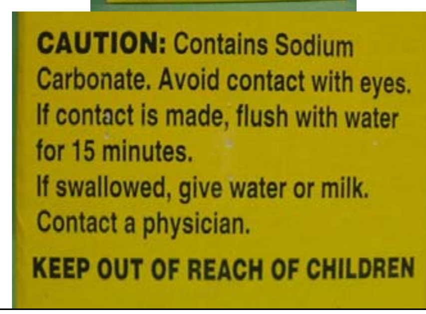
Potassium Bicarbonate and Sodium Bicarbonate[a]





[a] Handling and safety precautions similar to potassium bicarbonate.

Table 2. Consumer Products Containing Identical/Similar New Decon Green® and Decon Green® Classic Ingredients (Continued)

Potassium Carbonate and Sodium Carbonate[a]	
  	 
[a] Handling and safety precautions similar to potassium carbonate.	

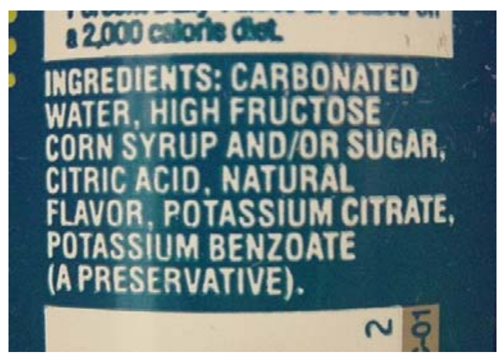
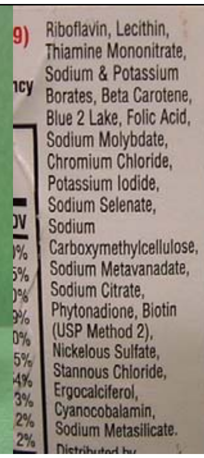
Potassium Citrate	
 	

Table 2. Consumer Products Containing Identical/Similar New Decon Green® and Decon Green® Classic Ingredients (Continued)

Sodium Molybdate[a]



[a] Handling and Safety precautions similar to potassium molybdate.

3.4 Reactor Tests

Table 3 gives stirred reactor data at room temperature (25 ºC) for New Decon Green® with conventional agents HD, VX, and GD. All of the agents are rapidly decontaminated to non-detectable levels, and exhibit the desired capacity of 1:50 agent to decontaminant.

Table 3. 25 ºC Reactor Data for New Decon Green®[a]

Time (min)	% HD			% VX			% GD		
10	4.5	3.1	4.7[b]	18.1	17.1	21.5	0.0	0.0	0.0
20	0.0	0.0	1.3	7.0	5.0	6.2	–	–	–
30	–	–	0.0	1.8	1.5	2.1			
40			–	0.3	0.2	0.6			
50				0.0	0.0	0.1			
60				–	–	0.0			

[a] 1 mL agent in 50 mL decon. Triplicate runs in stirred reactors. Results expressed as % agent remaining.
[b] Sampled at 5 min.

3.5 High Temperature Performance

Table 4 gives stirred reactor data at 50 ºC (122 ºF). The reaction rates are enhanced at the higher temperature such that all three agents are below detectable limits within 10 min. Thus, New Decon Green® is not only capable of decontaminating agents at 50 ºC, its efficacy is actually increased.

Table 4. 50 ºC Reactor Data for New Decon Green® [a]

Time min	% HD			% VX			% GD		
10	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
20	-	–	–	–	–	–	–	–	–
30									
40									
50									
60									

[a] 1 mL agent in 50 mL decon. Triplicate runs in stirred reactors. Results expressed as % agent remaining.

3.6 Low Temperature Performance

Table 5 gives stirred reactor data at 10 ºC (41 ºF). The reactions are slower than those at room temperature, with GD requiring up to 30 min to react, and VX and HD taking more than an hour.

Table 5. 10 ºC Reactor Data for New Decon Green® [a]

Time min	% HD			% VX			% GD		
10	9.1	26.3	27.2	23.4	27.2	23.0	2.6	3.3	4.5
20	6.7	3.4	1.8	15.2	14.7	18.2	0.2	0.3	0.2
30	8.7	0.7	0.2	10.3	11.2	11.1	0.0	0.0	0.0
40	1.3	0.3	0.2	6.2	7.2	8.3	-	-	-
50	0.7	0.3	0.2	4.2	5.2	5.1			
60	0.6	0.2	0.2	2.3	1.3	3.7			

[a] 1 mL agent in 50 mL decon. Triplicate runs in stirred reactors. Results expressed as % agent remaining.

3.7 Pot-Life

Owing to its greater stability compared to Decon Green® Classic, the pot-life of New Decon Green® has increased to at least 12 hr. Table 6 shows reactor data for HD, VX, and GD at 6 and 12 hr after mixing the decontaminant.

The data in Table 6 clearly shows that efficacy is maintained for at least 12 hr for HD, VX, and GD, suggesting that the pot-life is at least 12 hr. However, it should be noted that for this exercise, the decontaminant was mixed, handled, and stored under clean, sterile lab conditions. The exclusion of dirt and contaminants is of utmost importance when handling peroxide-based decontaminants as any impurities can severely hasten peroxide decomposition and, hence, loss of efficacy for the decontaminant. As under field conditions the cleanliness of ancillary decon equipment (buckets, mixing-tanks, blivets, etc.) cannot be guaranteed, it is

imperative that mix-on-the-fly applicators be developed and utilized to the greatest extent possible. This has been achieved and demonstrated for New Decon Green® with the development of the Intelagard® Decon Green-Specific Falcon™ (see below) for large-scale decontamination. Similar man-portable devices should also be developed as even a backpack type sprayer such as the Intelagard® MACAW™ (see below) will eventually become contaminant-ridden after repeated use and thus detrimental to the longevity of peroxide-based decontaminants. The current 5 gal New Decon Green® Kit does come with its own clean bucket, but post-mixing contamination of the decontaminant is practically assured as a result of wind-blown debris. Moreover, additional contaminants will be unavoidably introduced into the decontaminant if a mop is used to apply it. Thus, mix-on-the-fly applicators are always the best choice, for both large- and small-scale applicators, as only then is the pot-life issue fully removed as a potential problem.

Table 6. 25 ºC Reactor Data for New Decon Green®[a] after 6- and 12-hr Ageing

6 hr Ageing									
Time min	% HD			% VX			% GD		
10	4.7	3.5	12.2	17.7	15.9	17.2	0.0	0.0	0.0
20	0.6	0.0	0.8	5.4	6.8	6.9	–	–	–
30	0.0	0.7	0.6	1.3	1.4	1.6			
40	0.6	0.0	0.6	0.3	0.3	0.4			
50	0.5	0.0	0.6	0.0	0.0	0.0			
60	0.7	-	0.0	-	-	-			
12 hr Ageing									
10	8.2	6.5	15.2	15.5	18.0	14.5	0.0	0.0	0.0
20	0.0	0.0	0.0	3.4	4.3	3.9	-	-	-
30	-	-	-	0.8	1.0	1.0			
40				0.1	0.3	0.2			
50				0.0	0.0	0.0			
60				-	-	-			

[a] 1 mL agent in 50 mL decon. Triplicate runs in stirred reactors. Results expressed as % agent remaining.

3.8 <u>Bio Decon Testing</u>

3.8.1 <u>Suspension and Glass Surface Decon of *B. anthracis* Spores and *Y. pestis* Cells</u>

The results for the decontamination of *B. anthracis* NNR1Δ1 spores on glass slides and in suspensions are shown in Figure 1. Within 15 min, New Decon Green® afforded a near 6-log kill (150 spores remaining), whereas Decon Green® Classic and bleach achieved at least 8-log kills (no viable spores detected).

The results for the decontamination of *B. anthracis* Ames spores on glass slides and in suspensions are shown in Figure 2. Within 15 min, New Decon Green®, Decon Green® Classic, and bleach achieved at least 8-log kills (no viable spores detected).

The results for the decontamination of *Y. pestis* cells on glass slides and in suspensions are shown in Figure 3. Within 15 min, New Decon Green®, Decon Green® Classic, and bleach achieved at least 8-log kills (no viable cells detected).

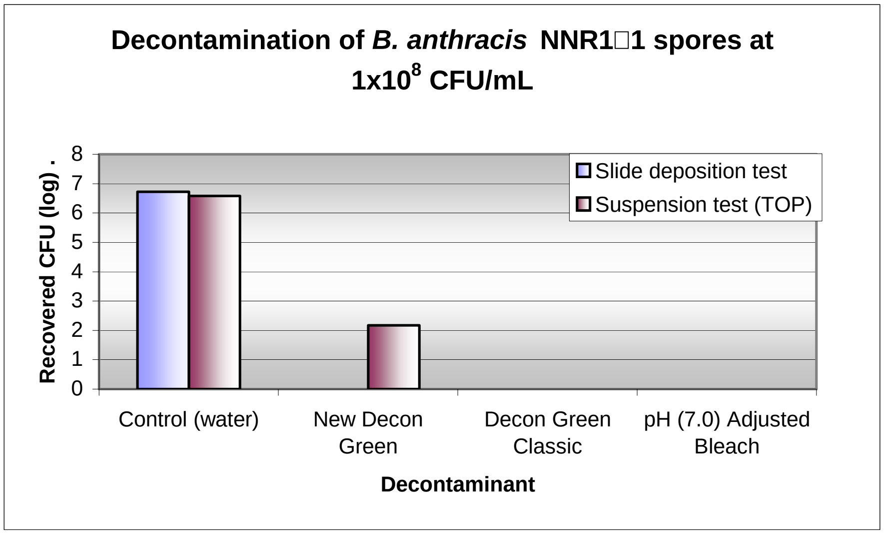


Figure 1. Viable 10^8 CFU mL^{-1} *B. anthracis* NNR1Δ1 Spores.
Exposed on glass slides and in suspensions for 15 min to New Decon Green®, Decon Green® Classic, and pH 7 Bleach.

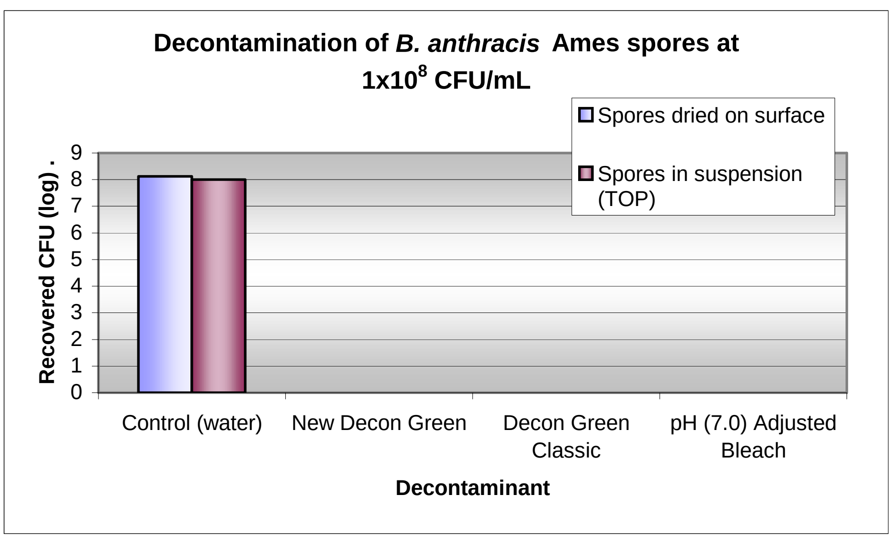


Figure 2. Viable 10^8 CFU mL^{-1} *B. anthracis* Ames Spores.
Exposed on glass slides and in suspensions 15 min to New Decon Green®, Decon Green® Classic, and pH 7 Bleach.

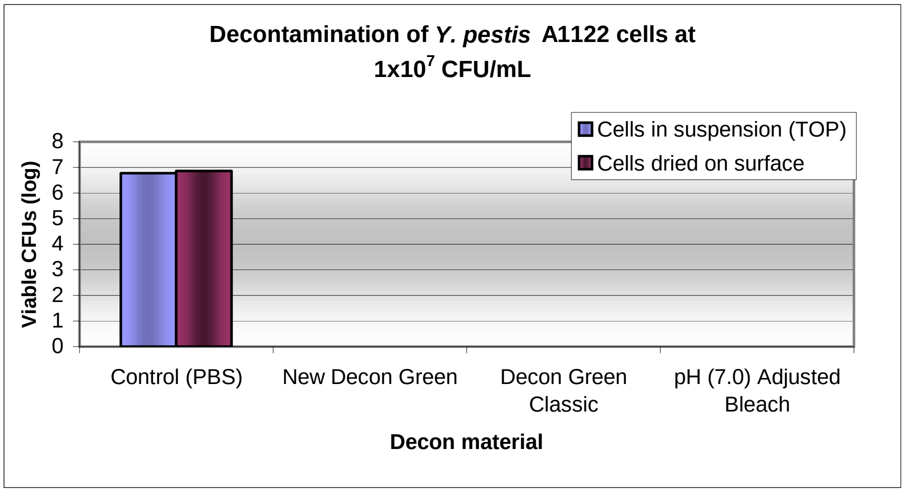


Figure 3. Viable 10^7 CFU mL^{-1} *Y. pestis* Cells.
Exposed on glass slides and in suspensions 15 min to New Decon Green®, Decon Green® Classic, and pH 7 Bleach.

3.8.2 Suspension Testing with *B. anthracis* and *B. atrophaeus* (Simulant) Spores

The suspension test is a useful test when screening a decontaminant for efficacy against biological agents. Prior to screening for efficacy versus *B. anthracis* spores on materials, it was desirable to determine a baseline indication as to the expected efficacy of the candidate decontaminants. Such information not only provides a means to ensure that the decontaminant is working according to the developer's expectations, but also provides necessary information as to the appropriate final resuspension volume to employ in the materials testing.

The suspension test results for *B. atrophaeus* spores shown in Figure 4 indicate that New Decon Green® affords a 6-log kill (32.7 average spores remaining) and Decon Green® Classic affords a 7-log kill (1.33 average spores remaining) in only 15 min.

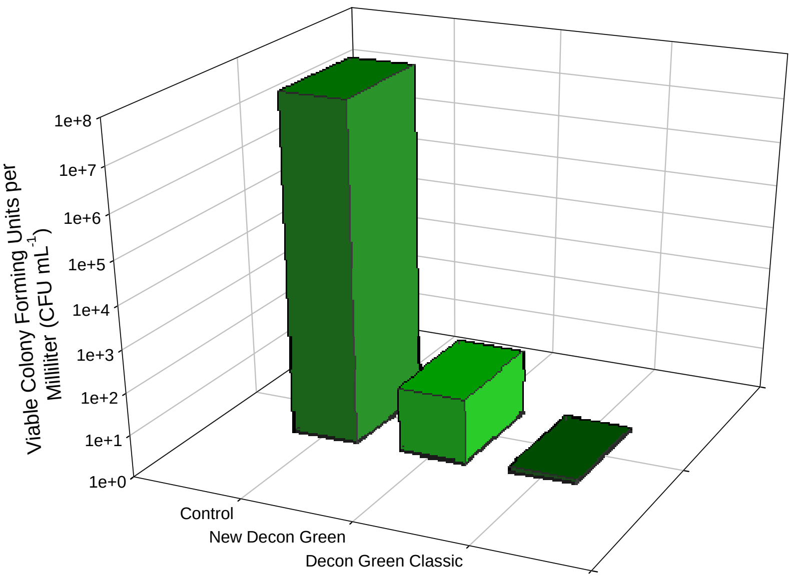


Figure 4. Viable 10^7 CFU mL^{-1} *B. atrophaeus* Spores. Exposed for 15 min to New Decon Green® and Decon Green® Classic.

3.8.3 *B. anthracis* on Rubber and CARC

A preliminary screen of the bio-efficacy of New Decon Green® and Decon Green® Classic was conducted using *B. anthracis* Ames spores and rubber material. For this initial test, ice cold LB broth and subsequent washes were used to neutralize the decontaminant activity. Results suggest that New Decon Green® is at least as effective as Decon Green® Classic in reducing the spore population on rubber (Figure 5, left column). New Decon Green® afforded at least a 6-log kill for Ames strain (2.67 average spores remaining) and >6-log kill for V1B strain (no viable spores detected). Decon Green® Classic yielded at least a 5-log kill for Ames strain (73 average spores remaining) and >6-log kill for V1B strain (no viable spores detected).

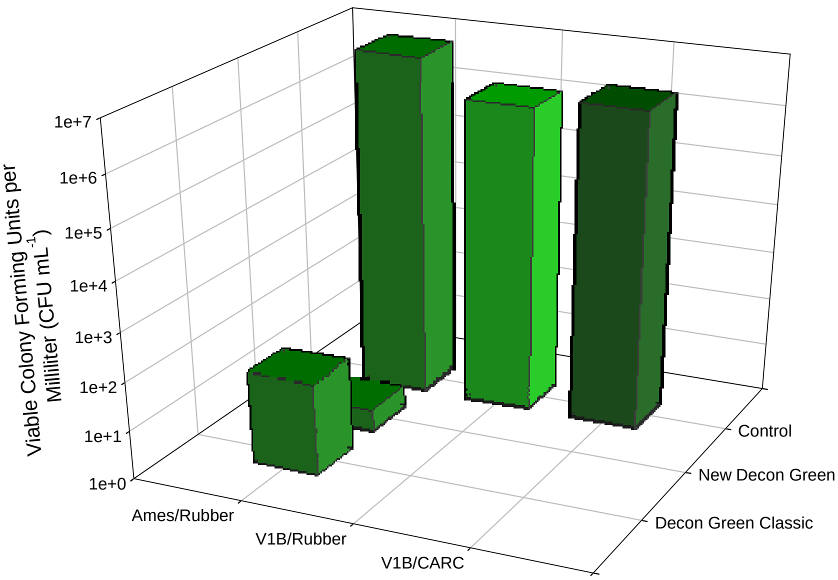


Figure 5. Viable ~0.5 cm^2 CARC and Rubber Coupons.
30-min exposure to New Decon Green® and Decon Green® Classic.
Left column: rubber coupons contaminated with 10^7 CFU *B. anthracis* Ames spores. Right columns: CARC and rubber coupons contaminated with 10^6 CFU *B. anthracis* Vollum 1B spores.

A side-by-side test was conducted to screen the efficacy of both New Decon Green® and Decon Green® Classic on *B. anthracis* Vollum 1B contaminated rubber and CARC. In this testing, the decontaminant was neutralized with 600 µL of 30% sodium metabisulfite at the end of the 30 min contact period. Initial results indicate that both formulations are effective in reducing the spore population on contaminated CARC and rubber samples (Figure 5, right columns). New Decon Green® and Decon Green® Classic afforded >6-log kills (no viable spores detected). Furthermore, the results suggest that there is no significant difference in the efficacy of either Decon Green formula on *B. anthracis* Vollum 1B spores on CARC or rubber materials.

Although it may appear that there exist differences in the *B. anthracis* strain resistance to New Decon Green® and Decon Green® Classic, such conclusions may not be drawn from the limited data presented in this report. It is possible that such differences may be attributable to the slightly different starting concentrations or due to the limitations of the assay. Further investigation into this issue is warranted and future studies are planned.

The results suggest that New Decon Green® has retained its efficacy against spore forming bacteria as compared to Decon Green® Classic. However, this decontaminant may not be labeled a sterilant for contact times of 30 min, based upon data collected during this initial screen for efficacy. More rigorous bio-efficacy testing is necessary to better characterize the contact time-spore kill kinetics and to characterize differences in strain resistance, if any. Such studies are planned.

It should be noted that biological agents (i.e. spores), unlike chemical agents, do not sorb into nor soften CARC paint, rubber, or other materials. Thus bio agents remain accessible to and unprotected from the decontaminant. Therefore, solvents which penetrate and soften the materials to be decontaminated (i.e. CARC paint) are not strictly necessary. Such is not the case for chemical agents, where penetrating, softening solvents *are necessary* for fast, efficacious decontamination of surfaces susceptible to softening by chemical agents (i.e. CARC paint, see below).

3.9 CARC Paint Softening

3.9.1 Softening by Agents

Before presenting the results of CARC panel testing in the next section, it is imperative to discuss the softening of CARC paint by chemical agents and its ramifications for decontamination. Such a discussion will allow the results of the panel tests to be easily understood. A diagram depicting the process of agent penetration into a material with concomitant softening is shown in Figure 6.

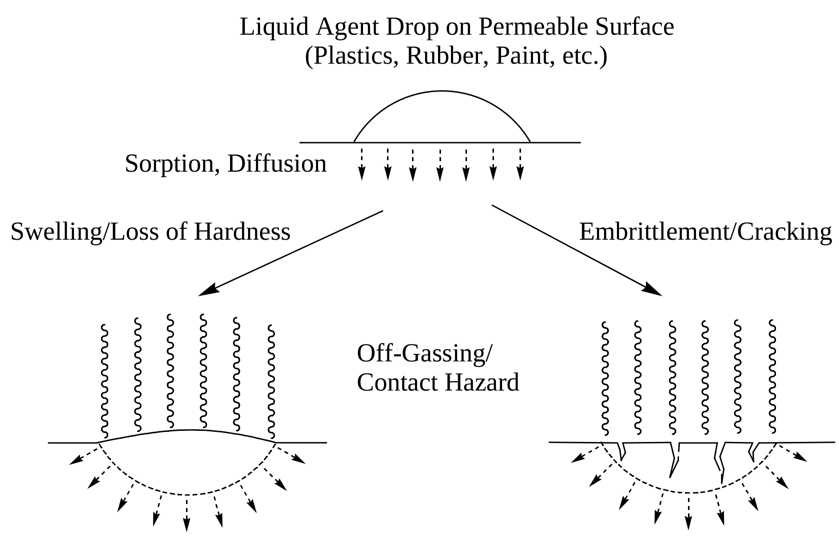


Figure 6. Agent Sorption into Susceptible Surface

Swelling and/or embrittlement of a material is often accompanied by its softening. Further, note that once the material is softened, off-gassing will occur, even after excess liquid agent is removed from the surface (as a result of washing with soapy water, for example).

To determine the ability of HD, VX, and GD to soften CARC paint, the same CARC-painted aluminum coupons used in panel testing (see below) were immersed into the agents for periods of up to 1 week. Such extended periods were necessary so that even subtle softening could be observed. Changes to the hardness were then assessed using a standard “pencil hardness gauge” or scratch/gouge test. The results are shown in Table 7.

It can be clearly seen that all three agents soften CARC paint, with HD being in a class by itself. VX is more effective at softening CARC paint than GD, which softens only minimally.

The ability of chemical agents to soften CARC paint will have a deleterious effect on the ability to decontaminate CARC paint. This effect is quite dramatic; being clearly evident in the Panel Test data presented in the next section.

Table 7. Softening of CARC Paint by HD, VX, and GD[a]

Time	HD	VX	GD
1 day	–	0	0
3 days	–	-2	-1
6 days	–	-2	–
7 days	-7	-	-1

[a]Panel completely immersed in agent at 25 ºC for the indicated time period. Zero means no change. Negative values denote softening, i.e. a softer pencil lead is able to scratch the affected surface.

3.9.2 Softening by Decontaminants

AR 70-71, "Nuclear, Biological, and Chemical Contamination Survivability of Army Material", defines two important criteria which directly impact decontamination. The first is "Decontaminability":

> The equipment must be capable of being decontaminated using standard NBC decontaminants and procedures available in the field, to the point that the contaminant poses no casualty-producing hazard to unprotected personnel exposed during normal mission profile of the equipment.

The second is "Hardness":

> "The equipment must be resistant to the materiel-damaging effects of NBC contaminants and the decontamination agents and procedures required to remove the contamination."

One can readily gather from the agent softening results mentioned above that CARC does not exactly exhibit "hardness" when it comes to HD, and the paint is even pushing this definition for VX.

CARC is already on more than a few vehicles that may eventually need to be decontaminated; nothing can be easily done about its lack of "hardness" at this point. So this leaves the issue, "decontaminability", to be considered next.

AR 70-71 requires that "mission essential equipment and materiel shall be hardened to ensure that degradation over a 30-day period of no more than 5% in selected

quantifiable essential characteristics is caused by five exposures to NBC contaminants, decontaminants, and decontaminating procedures encountered in the field". To determine if CARC paint could stand up to five decontamination cycles, panel tests were performed, without agents. These results are shown in Table 8.

Table 8. CARC Hardness Changes after 5-Decon Cycles[a]

Decon Cycle	DG Classic	New DG
#1	0	0
#2	-2	-2
#3	-4	-2
#4	-4	-2
#5	-4	-4

[a] Zero means no change in hardness. Negative values denote softening, i.e. a softer pencil lead is able to scratch the affected surface.

Previous CARC testing with Decon Green® Classic and New Decon Green® deemed a hardness change of 2, either softening or hardening by two classes, as being acceptable.[9] Using this criteria, the results in Table 8 reveal that CARC paint can withstand two decontamination cycles with Decon Green® Classic before excessive softening occurs, whereas it can take four cycles with New Decon Green®. Neither decontaminant permits the 5-decontamination cycles of CARC called out by AR 70-71. Yet some softening is unavoidable to be able to quickly decontaminate agents to acceptable levels as discussed in the next section.

3.10 Panel Tests

As a starting point to show the ease of decontamination of "hard", impermeable surfaces, in contrast to "soft" CARC paint surfaces, Table 9 gives results for Decon Green® Classic decontamination of bare aluminum panels contaminated with HD, VX, and TGD. Also indicated in the tables are the required Operational Decontamination Levels for each agent.

Table 9 shows how easy it is for Decon Green® Classic, and probably many other decontaminants, to decontaminate a non-sorptive surface, aluminum metal, in this instance. Both TGD and HD are decontaminated (within 15 min) to well below their required Operational Decontamination Levels. Although VX is actually decontaminated to more than an order of magnitude lower than TGD and HD (≥99.99 %), for Decon Green® Classic the remaining total amount of VX (0.0999 $\mu g/cm^2$) is higher than the required level (0.078 $\mu g/cm^2$). However, New Decon Green® decontaminates VX to an even lower level (0.0335 $\mu g/cm^2$), which is less than half the required level. Thus, on hard, non-sorptive surfaces the decontamination of TGD, HD, and VX to the required operational levels is easy and straightforward. However, on sorptive materials, such as CARC paint (see below), decontamination to these required levels becomes difficult, perhaps impossibly so for VX.

Table 9. Bare Aluminum Panels Decontaminated by Decon Green® Classic and New Decon Green® [a]

Agent	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
TGD with DG® Classic	0.347	0.475	0.174	0.996	99.90
TGD with New DG® [d]	0.500	0.449	0.055	1.004	99.90
Req. Level	1.67				
HD with DG® Classic	0.49	0.41	0.022	0.922	99.91
HD with New DG® [d]	0.52	0.46	0.026	1.01	99.90
Req. Level[c]	10.0				
VX with DG® Classic	0.0169	0.000	0.083	0.0999	99.990
VX with New DG®	0.0015	0.000	0.032	0.0335	99.997
Req. Level[c]	0.078				

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Level required for Operational Decontamination.
[d] Developmental formula.

Tables 10-14 give results for New Decon Green® (New DG®), Decon Green® Classic (DG® Classic), and DF200 decontamination of CARC panels contaminated with HD, THD, VX, TVX, and TGD. For these tests, DF200 was applied as a liquid.

It is readily apparent that New Decon Green® is not as effective as Decon Green® Classic at decontaminating HD on CARC paint as evidenced by both the higher contact hazard and residual hazard. Also, the amount of the original agent actually destroyed is considerably less for the new formula. Regarding the level of decontamination required for Operational Decontamination, 10.0 μg/cm^2 (or 100 mg/m^2), only Decon Green® Classic comes close to attaining this level after a 15 min decontamination time, whereas New Decon Green® is more than four times the level. The level provided by DF200 is an order of magnitude higher than the prescribed level. Concerning the amount of agent actually destroyed during the 15 min contact time, Decon Green® Classic achieves 92.2% destruction, New Decon Green® 79.0%, and DF200 62.9%. Thus, both Decon Green® formulas far exceed the decon efficacy of DF200 for HD on CARC paint. The performance of each of the three decontaminants for the decontamination of HD on CARC paint is perfectly understandable, given the nature and amount of solvent in each. The reason for their behavior is discussed below.

Table 10. Decontamination of HD on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
New DG™	43.3	12.26	154.6	210.16	79.0
DG™ Classic	17.2	3.56	57.0	77.76	92.2
DF200[c]	101.5	22.61	247.2	371.31	62.9
Req. Level[d]	10.0	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Envirofoam Technologies.
[d] Level required for Operational Decontamination.

Table 11. Decontamination of THD on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
New DG®	115.09	12.97	29.9	157.96	84.2
DG® Classic	16.38	2.65	4.8	23.83	97.6
DF200[c]	536.12	24.86	91.3	652.28	34.8
Req. Level[d]	10.0	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Envirofoam Technologies.
[d] Level required for Operational Decontamination.

Again, the results show the same trend as observed for (unthickened) HD: Decon Green® Classic performs better than New Decon Green® which performs far better than DF200. Decon Green® Classic presents a 16.38 μg/cm^2 HD contact hazard level after the 15 min decontamination period, which is still close to the required Operational Level of 10 μg/cm^2, whereas the value afforded by New Decon Green® (115.09 μg/cm^2) is now over 11 times higher. Even more notable is the level seen for DF200 (536.12 μg/cm^2), which is now more than 50 times the Operational Level. As for overall destruction of the thickened HD, Decon Green® Classic and New Decon Green® attain 97.6% and 84.2%, respectively, which is an improvement over their performance with unthickened HD (92.2 and 79.0%). However, DF200 performs much worse for thickened HD, destroying only 34.8% (vs. 62.9% for unthickened HD).

The remarkable differences in the data for thickened and unthickened HD shed light on what is contributing to the different behavior of the decontaminants. It is known that HD sorbs into and softens CARC paint (see above). Thickened HD, although more persistent, is slower to penetrate CARC paint due to both its slower spreading rate and the competition of the thickener to retain the HD. Thus, not as much of the HD penetrates and softens the CARC paint. This lessened softening ability of thickened HD for CARC paint allows for more thorough

decontamination, with one important caveat: the decontaminant must be able to quickly dissolve and penetrate the thickener to access and react with the HD.

Regarding Decon Green® Classic, the contact levels achieved for thickened and unthickened HD are remarkably similar, 16.38 and 17.2 $\mu g/cm^2$ (15 min) and 2.65 and 3.56 $\mu g/cm^2$ (45 min), indicating that this decontaminant does a good job at dissolving the thickener. However, the residual amount remaining for the thickened HD is dramatically decreased compared to the unthickened HD: 4.8 vs. 57.0 $\mu g/cm^2$, due to the inability of the thickened HD to penetrate and soften the CARC paint.

As for New Decon Green®, it is immediately apparent that it does not dissolve the thickener as well since the 15 min contact levels achieved for thickened and unthickened HD are starkly different: 115.09 and 43.3 $\mu g/cm^2$. It should be noted that HD and other agents are thickened with plastics such as polymethylmethacrylate (PMMA). The low-solvent- containing New Decon Green® does not dissolve/soften plastic as well, including "heavily contaminated plastic", i.e. thickened agent. Some HD is undoubtedly leached and destroyed from the thickened HD residing on top of the CARC panel, as evidenced by the pasty, but tacky nature of the remaining film. Owing to its reduced HD content, the remaining thickened HD is perhaps more aptly described as "HD-plasticized PMMA". The fact that the 45 min contact times remain similar is easily understood by the fact the bulk of the remaining "HD-plasticized PMMA" on the CARC panel is removed during the initial 15 min contact test as it tends to stick to the latex panel employed for this test (see Experimental). Again, because of the inability of thickened HD to quickly penetrate and soften CARC paint, the residual HD remaining in the CARC paint is much less for thickened HD (29.9 $\mu g/cm^2$) than for unthickened HD (154.6 $\mu g/cm^2$).

The same arguments hold true for the DF200 results. The huge increase in the 15 min contact time for thickened HD, 536.12 $\mu g/cm^2$ (representing more than half of the applied 1000 $\mu g/cm^2$ HD, by the way), compared to 101.5 $\mu g/cm^2$ for unthickened HD, is simply due to the inability of DF200 to dissolve plastic-thickened HD/HD-plasticized PMMA plastic. Again, the second 45 min contact time remains comparable between thickened and unthickened HD, 24.86 and 22.61 $\mu g/cm^2$, as the remaining HD-plasticized plastic film on the CARC panel is entirely removed by the latex film during the first 15 min contact assessment.

As can be seen by the results for thickened and unthickened HD, a "double-penalty" has occurred as a result of reducing the penetrating solvent content to make the Decon Green® Classic formula more material friendly to CARC paint, the plastic HMMWV light housing, and the plastic M40 Mask Lens. Penalty number one is that the new formula does not penetrate and soften CARC paint as well as to afford as thorough a decontamination of the sorbed HD. Penalty number two is that the new formula does not dissolve thickener/plastics as well as to afford as thorough a decontamination of thickened HD/HD-plasticized plastic. These data raise two important questions: 1) What is more important, to protect contaminated paint or to decontaminate it? 2) What is more important, to protect plastics or to decontaminate plastics and HD thickened with plastic? Such is the dilemma faced in both the decontamination of soft materials and the decontamination of thickened agents.

For VX, again Decon Green® Classic performs the best, yielding the lowest contact hazards, 1.46 (15 min) and 0.37 μg/cm^2 (45 min); the lowest residual agent, 47.2 μg/cm^2; and the best, overall decontamination, 95.1%. New Decon Green®, is worse, with contact hazards of 4.14 and 1.28 μg/cm^2; residual agent of 53.8 μg/cm^2; but only a slightly lower overall decontamination of 941%. Still worse is DF200, affording contact hazards of 8.28 and 3.17 μg/cm^2; residual agent 109.3 μg/cm^2; and a significantly worse overall decontamination of 87.9%.

Table 12. Decontamination of VX on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
New DG®	4.14	1.28	53.8	59.22	94.1
DG® Classic	1.46	0.37	47.2	49.03	95.1
DF200[c]	8.28	3.17	109.3	120.75	87.9
Req. Level[d]	0.078	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Envirofoam Technologies.
[d] Level required for Operational Decontamination.

These results are easily understood using similar arguments presented above for HD. VX penetrates and softens CARC paint, but not to such a degree as HD (see below). Although less residual agent remains in the paint for all three decontaminants, a penetrating, softening solvent is still required as evidenced by the large amount of residual VX left by DF200. Furthermore, additional evidence for the importance of high solvent/low water content is apparent from the observed DF200 15 min contact hazards: 101.5 μg/cm^2 for HD vs. only 8.28 μg/cm^2 for VX. Thus DF200, which has the lowest solvent content of the three decontaminants, is able to more easily dissolve water-soluble VX than water-insoluble HD.

The required Operational Decontamination Level for VX (0.078 μg/cm^2) is more than three order of magnitudes lower than that of HD (10.0 μg/cm^2); values no doubt reflecting the extreme difference in their relative toxicities. Decon Green® Classic at least comes close to reaching the desired level for HD, attaining a 17.2 μg/cm^2 15 min contact level. And although the value of 1.46 μg/cm^2 attained for VX in the 15 min contact test is an order of magnitude lower than that of HD, it is still more than one order of magnitude higher than the required Operational Level. So although VX does not penetrate and soften CARC paint to the same extent as HD (see below), it remains the most difficult to decontaminate simply because of the low operational level one needs to attain.

For thickened VX, the best decontaminant is not as clear cut as for unthickened VX. While Decon Green® Classic still affords the lowest contact hazards, 1.9 (15 min) and 0.29 μg/cm^2 (45 min), the residual contamination of 33.4 μg/cm^2 is higher than that afforded by DF200 (23.4 μg/cm^2). Their overall decontamination achievements of 96.4 and 96.6% are

virtually identical. Most of the gains observed for DF200 for thickened VX vs. unthickened VX arise from the decreased residual VX, which is sorbed into the CARC paint. As pointed out above for thickened HD, thickened VX is likewise slower to soften CARC paint; thus it is more accessible to reaction with non-penetrating DF200. The data further show that thickened, water-soluble VX is easier for DF200 to dissolve and react with than thickened, water-insoluble HD, as the contact hazard observed for thickened and unthickened VX are nearly the same. The results for New Decon Green® for thickened and unthickened VX are virtually identical, indicating that it likewise dissolves thickened VX. The fact that no improvement is seen for thickened VX suggests that the reaction capacity may have been reached for New Decon Green®. Thus, although the thickened VX was more accessible, not sorbed into the paint to such a degree, it was simply not fully reacted with within the 15 min decontamination time. It should be pointed out that both Decon Green® Classic[1] and DF200[12] provide for faster reactions with VX as shown by the reactor studies (see above).

Table 13. Decontamination of TVX on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
New DG®	4.8	1.31	54.2	60.31	94.0
DG® Classic	1.9	0.29	33.4	35.59	96.4
DF200[c]	8.7	2.09	23.4	34.19	96.6
Req. Level[d]	0.078	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in $\mu g/cm^2$. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Envirofoam Technologies.
[d] Level required for Operational Decontamination.

For thickened GD, Decon Green® Classic performs only slightly better than New Decon Green® and DF200 as both the 15 and 45 min contact hazards are lowest, 5.2 and 1.66 $\mu g/cm^2$; the residual hazard is lowest, 20.24 $\mu g/cm^2$; and the overall decontamination is slightly higher at 98.0%. As pointed out earlier, GD does not soften CARC paint nearly as much as HD and VX, and thickened GD is even less able to do so; thus the similarity of the residual contact hazards presented by all three decontaminants. However, a slight trend based on the penetrating ability of the decontaminant is still in evidence, with Decon Green® Classic yielding the lowest residual hazard, 13.38 $\mu g/cm^2$; followed by New Decon Green®, 14.9 $\mu g/cm^2$; and then DF200, 16.20 $\mu g/cm^2$. The fact that Decon Green® Classic provides for the lowest contact hazard shows that it is still the leader in dissolving thickened agent, TGD being no exception.

Table 14. Decontamination of TGD on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
New DG®	9.1	1.84	14.9	25.84	97.4
DG® Classic	5.2	1.66	13.38	20.24	98.0
DF200[c]	8.2	1.95	16.20	26.35	97.4
Req. Level[d]	1.67	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Envirofoam Technologies.
[d] Level required for Operational Decontamination.

The Operation Decontamination Level required for GD is 1.67 μg/cm^2**.** The 15 min contact hazard level afforded by the best performer, Decon Green® Classic, at 5.2 μg/cm^2 is at least within an order of magnitude of this value, as are New Decon Green® (9.1 μg/cm^2) and DF200 (8.2 μg/cm^2). Thus, unlike VX, it may be quite possible with further tinkering to attain the required Operational Decontamination Level for GD.

3.11 Decon of Oily Surfaces

Besides affording enhanced decontamination of soft surfaces such as CARC, decontaminants possessing good solvency penetrating ability also excel at decontaminating oily surfaces. Table 15 shows results for the decontamination of CARC paint laced with diesel fuel. HD was chosen to illustrate the relative effectiveness of Decon Green® Classic, New Decon Green® and DF200, as it is the most difficult agent to decontaminate.

Table 15. Decontamination of HD on CARC Paint[a] in Diesel Fuel Presence (Oily Surface Decon)

Decon	Diesel Applied Before HD[b]			Diesel Applied After HD[c]		
	C1	C2	Extract	C1	C2	Extract
DGC	45.9	25.82	177.4	52.41	26.30	294.2
NDG	59.9	33.27	220.6	55.69	36.22	320.0
DF200[d]	66.1	34.18	247.3	47.57	30.84	390.1

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard (C1, 15 min; C2, 45 min) and Residual agent (Extract) expressed in μg/cm^2. Average of six replicates reported.
[b] Diesel applied to panel prior to contamination with HD.
[c] Diesel applied to panel following HD application and 1 hour dwell time.
[d] Envirofoam Technologies.

The results reveal that diesel renders decontamination of HD on CARC more difficult as Decon Green® Classic is now unable to reduce either the contact hazard or residual

hazards to the lowest levels noted above for “oil-free” CARC. Indeed, its performance is now only slightly better than New Decon Green®. Especially note the difference between adding diesel before or after the HD. For diesel added after HD contamination, the residual hazards are particularly high, suggesting that diesel acts as a protective barrier to prevent decontaminants from reaching the HD. To a lesser extent, this is also true for HD applied after diesel, where the residual hazards are lower, but still higher than in the absence of diesel (see above).

3.12 Further Comments on Decontamination of CARC Paint

Once sorbed into CARC paint, agent is impervious to aqueous solutions such as soapy water and even bleach as water cannot soften nor penetrate the paint to attack the sorbed agent. However, solvents which can soften CARC paint are able to penetrate the paint to attack sorbed agent; thus providing a greatly improved decontamination. These processes are depicted in Figure 7.

Penetrating decontaminants, e.g. DS2 and Decon Green®, sorb right into the material just like the agents (refer to Figure 3, above), depleting the sorbed agent, resulting in greatly-diminished contact and off-gassing hazards. Non-penetrating decontaminants such as HTH, STB and bleach, and even soapy water, are limited to just removing excess liquid agent from the surface, letting the sorbed agent remain as a substantial contact, and off-gassing hazard. Of course, penetration of the material by a decontaminant can result in additional softening/cracking/embrittlement beyond that caused by the agent itself. However, this additional damage may be an acceptable trade-off when the costs of prolonged off-gassing and contact hazard on soldier health and the continued burden of having to wear cumbersome protective gear are considered.

3.13 Comments on Off-Gassing

Off-gassing or vapor hazard is another test used to determine the efficacy of a decontaminant. This test purports to answer the question: to what extent does a contaminated surface contaminate the air around it?

ORD documents[13] specify the desired vapor/aerosol levels for Nerve-G, Nerve-VX, and Blister-H agents as reproduced in Table 16. The rationale behind this requirement is to “allow resources to be returned to operational use and reduce the MOPP level required for personnel.” With regard to the detection of agent on contaminated surfaces the ORD further mentions the use of “detectors measuring from a distance of 1 in. from the surface . . ..” Thus, it appears that vapor concentration quite close to the surface is of primary interest as this is where testing is to be done under real-world field conditions.

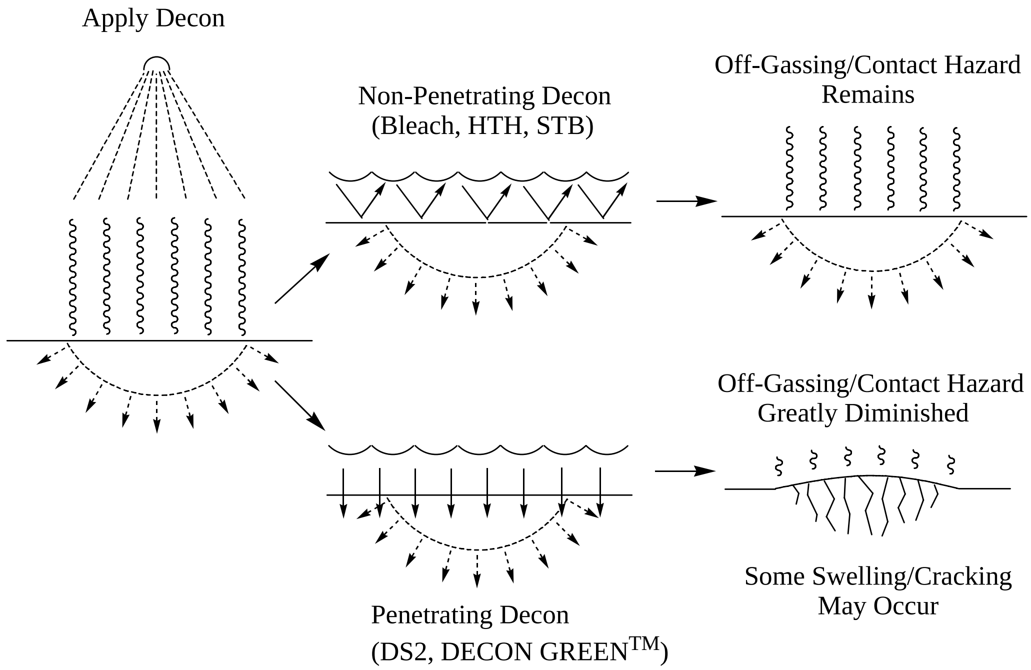


Figure 7. Penetrating vs. Non-Penetrating Decontamination of Agent Sorbed in Surface

Table 16. ORD Vapor/Aerosol Levels (mg/m^3)[13]

Level	Nerve-G	Nerve-VX	Blister-H
Threshold (30 min, Cumulative)	<0.001	<0.001	<0.02
Objective (8 hr, Time Weighted Average)	<0.0001	<0.00001	<0.0003

To rationalize exactly what a vapor concentration presumably emanating from a contaminated surface means, it is fortunate that the ORD mentions a measuring distance of 1 in. from the surface. For example, one would not expect to find the same vapor concentration at distances of 1 in. and 100 yd from the surface. So it is the concentration very near the surface that is of prime concern, with the understanding that it would tend to decrease as one gets further away.

As mentioned above, none of the agents, GD, VX, HD, could be decontaminated from CARC paint to below the contact hazard levels specified by ORD documents, 16.7, 0.78, 100 mg/m^2. Thus, these values serve as a useful starting point when considering off-gassing or vapor hazard from a contaminated surface.

It is easy to envision toxic vapor arising from a contaminated surface as depicted in Figure 8. For purposes of simplification, zero-wind condition is assumed so that the “cloud” is symmetric.

Perhaps a particularly illustrative way to relate a known *surface* concentration hazard to an anticipated *vapor* concentration hazard is to consider a shaft of air over a unit area near the center of the contaminated area (to avoid edge-effects) as shown by the square in Figure 8, and the height the vapor concentration of a critical hazard level (Table 16) can be expected to extend or rise.

The contact and vapor hazard levels specified by the ORD are expressed in mg/m^2 and mg/m^3, which is the convenient unit to use when considering larger items, anywhere from a vehicle to a contaminated urban area. Yet, it is more convenient for small items such as a rifle or the small 2 in. D laboratory panels utilized in this work to represent the contact hazard in $\mu g/cm^2$. However, whether one considers a 1 m^2 or 1 cm^2 shaft area, the distance or height to which a particular surface contamination can extend (ignoring edge effects) is the same. Sample calculations for GD (the worst case) are shown in Table 17 for the ORD contact exposure levels and the threshold vapor levels of Table 16. Illustration of the relative heights to which the vapor hazard could theoretically possibly rise over larger deposits is shown in Figure 9 (smaller items are discussed below). Note that vertical dimensions of the shafts in Figure 9 (in ft) are not to scale with the bases (in m).

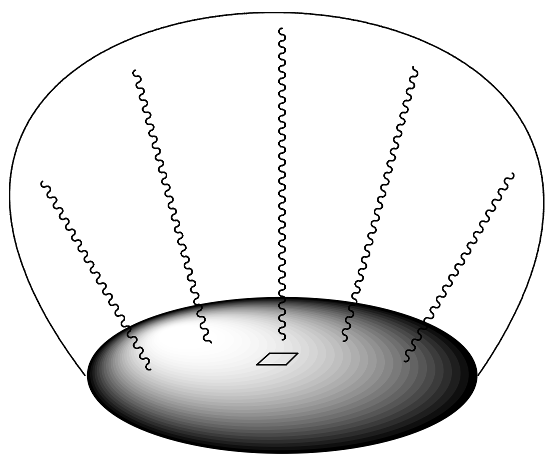


Figure 8. Off-Gassing from Contaminated Surface and Associated Vapor Cloud under Zero-Wind Conditions

Table 17. Height Calculations for GD Threshold Vapor Hazard Concentration from Surface Possessing GD ORD Contact Exposure Level

	Shaft-Base Dimension	
	1 m × 1 m	1 cm × 1 cm
Amount of GD contained in shaft base	16.7 mg	1.67 μg
Volume of air required to yield vapor concentration of 0.001 mg/m^3	16.7 mg ÷ 0.001 mg/m^3 = 16,700 m^3	1.67 μg ÷ 0.001 mg/m^3 × (1 mg/1000 μg) = 1.67 m^3
Height of shaft required to accommodate vapor hazard volume of V ÷ A = H	16,700 m^3 ÷ 1 m^2 = 16,700 m or 54,900’ [a]	1.67 m^3 ÷ 1 cm^2 × $(100\ cm/m)^2$ = 16,700 m

[a] 5280’ = 1 mile = 1.609 km

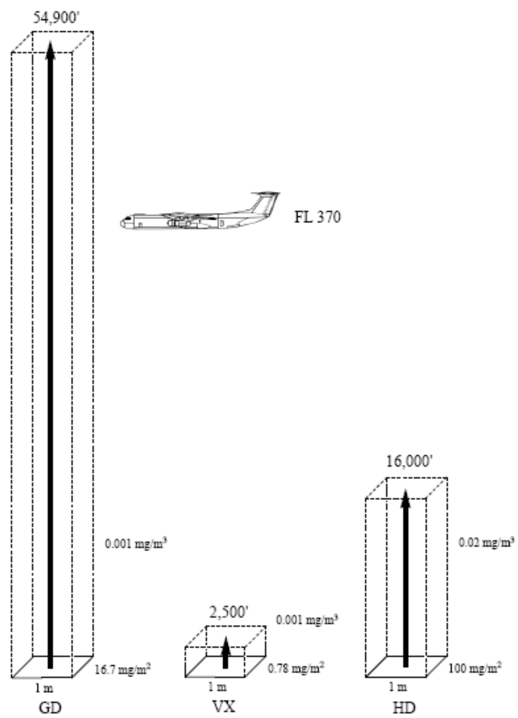


Figure 9. Height of Potential ORD Threshold Vapor Contamination Levels (mg/m^3) Arising from Surface with Known ORD Contact Hazard Contamination Level (mg/m^2) for GD, VX, and HD (see text).

It is clear from Figure 9 that at threshold contact hazard levels, the potential vapor hazard levels for each agent extend quite far from the surface. GD, for example, extends upward beyond the typical flight levels (FL) of passenger jets. And the situation is even more absurd if the objective vapor hazard levels are considered: In this instance, GD would rise to 167 km, albeit still comfortably below the space station (360 km). Thus, it is quite obvious from Figure 9 that at a height of 1 in. above the surface, vapor hazard levels resulting from the ORD contact hazard levels should be quite enormous.

Even though small items would not generate the outrageous plume associated with large vehicles or contaminated wide-areas (Figure 9), they can still contaminate quite large volumes of air when taken into vehicles, aircraft, buildings, etc. For example, take the interior volume of the C-141 cargo plane shown in Figure 9 which is about 1000 m^3. To exceed the threshold vapor hazard concentration for GD of 0.001 mg/m^3 within this aircraft would require just a *single item less than the size of a small book* contaminated at the GD contact hazard level of 16.7 mg/m^2 being brought on board (an item possessing an area of about 600 cm^2). So similar arguments, regarding the futility of bothering to measure vapor hazards when the known contact hazard is in excess of the ORD, apply to small items as long as these items are still able to contaminate large volumes of air within their vicinity or when brought into confined areas. Thus, it is under significant contact hazard level that it is safe to say that any off-gassing agent concentrations would be very high compared to even the threshold ORD vapor levels—sky high.

As previously presented above, detectable contact hazard levels for VX, GD, and HD are typically present in excess of the ORD contact exposure levels following the decontamination of CARC paint, even with the best of decontaminants. Nevertheless, vapor hazard assessments were performed for GD and HD following decontamination with New Decon Green®. These results are summarized in Table 18. Note that the results have been normalized to a 1 cm^2 surface area to allow direct comparison of different size panels.

Starting with the total amount of GD (0.0020) and HD (0.053 mg/cm^2) off-gassing during the 12 hr collection period, these amounts are about an order-of-magnitude lower than the nominal amounts expected to still be on the panels following decontamination (0.026 and 0.21 mg/cm^2). Thus, it is not clear whether the discrepancy arises from unavoidable losses during the prolonged collection period, sample tube trapping-efficiency/capacity, incomplete evaporation of agent from the CARC panels, or some combination of these. Additional work is required to sort this out and to get a mass-balance to account for all of the agent.

Table 18. Off-Gassing for GD and HD on CARC Following Decontamination with New Decon Green® [a]

Agent	Nominal Total Agent Remaining on Panel[b] (mg/cm^2)	Total 12-hr Off-Gassing Amount (mg/cm^2)	12-hr Off-Gassing Concentration ($mg/cm^2/m^3$)	ORD Threshold Vapor Level[c] (mg/m^3)
GD	0.026	0.0020	0.0093	<0.001
HD	0.21	0.053	0.247	<0.02

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Vapor sampled at 300 mL/min for 12 hr. Results expressed in $\mu g/cm^2/m^3$. Average of six replicates reported.
[b] From Tables 10 and 14.
[c] Ref. 13.

Considering the area-normalized off-gassing concentrations for GD and HD, 0.0093 and 0.247 $mg/cm^2/m^3$, respectively, these concentrations are about 10-times the ORD vapor threshold levels (0.001 and 0.02 mg/m^3). Of course, without normalization to the 20 cm^2-area of the panels, the off-gassing concentrations would be 20-times higher (0.19 and 4.94 mg/m^3), or about 200-times the ORD levels. Of course, the problem is which value is correct: the area-normalized value or the higher total-panel value? To add even more confusion, if the results had been obtained at different flow rates, say 30 mL/min or 3 L/min, rather than the 300 mL/min employed, the results would be 10-times more concentrated for the former, yet 10-times less for the latter. Thus, the current vapor hazard test procedure is riddled with ambiguities and its interpretation is not as sound and straightforward as the rigorous, completely unambiguous contact hazard test. Better procedures and assumptions need to be made before meaningful results can be obtained to truly assess the vapor hazard arising from surfaces of various dimensions and contamination levels. In the meantime, contact hazard and/or residual (extraction) tests remain the only reliable indication of decontamination efficacy.

3.14 Cold and Arctic Weather Type (CA^2WT) Decon Green®

Because of the tendency of bicarbonate ($KHCO_3$) ingredient of New Decon Green® to precipitate at temperatures below freezing, the "Cold and Arctic Weather Type" Decon Green® or CA^2WT DG was developed using carbonate (K_2CO_3) instead. With this modification, precipitation does not occur at temperatures above –15 ºC (5 ºF). Yet owing to the stabilizing effect of citrate, CA^2WT DG possesses long-term stability.

Tables 19-21 show the results of CARC panel tests for HD, VX and TGD decontamination, comparing CA^2WT DG to New Decon Green® and Decon Green Classic®. The efficacy of CA^2WT DG is quite similar to that of New Decon Green® as the identical solvent system is employed; Decon Green Classic® remains superior to both.

Table 19. CA^2WT DG Decontamination of HD on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
CA^2WT DG	30.3	13.50	116.6	160.4	84.0
New DG®	27.7	12.34	103.3	143.34	85.7
DG® Classic	10.7	3.87	46.5	61.07	93.9
Req. Level[c]	10.0	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Level required for Operational Decontamination.

Table 20. CA^2WT DG Decontamination of VX on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
CA^2WT DG	14.1	10.56	107.3	131.96	86.8
New DG®	12.6	7.12	88.8	108.52	89.1
DG® Classic	5.6	1.53	56.9	64.03	93.6
Req. Level[c]	0.078	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Level required for Operational Decontamination.

Table 21. CA^2WT DG Decontamination of TGD on CARC Panels[a]

Decon	Contact 15 min	Contact 45 min	Residual (Extraction)	Total Agent Recovered	% Decon[b]
CA^2WT DG	6.35	0.95	4.96	12.26	98.8
New DG®	4.37	0.43	3.45	8.25	99.2
DG® Classic	1.75	0.28	2.04	4.07	99.6
Req. Level[c]	1.67	–	–	–	

[a] Initial contamination level: 10 g/m^2. Agent dwell time: 1 hr. Decontamination time: 15 min. Contact hazard and Residual agent expressed in μg/cm^2. Average of six replicates reported.
[b] Percent of applied agent destroyed.
[c] Level required for Operational Decontamination.

Table 22 shows stirred-reactor tests for CA^2WT DG with HD, VX, and GD at 25 and 10 ºC. These results show that CA^2WT DG reactivity is somewhat slower than New Decon Green® at 25 ºC, but affords comparable reactivity at 10 ºC. Counterintuitively, CA^2WT DG appears to afford somewhat faster reactivity towards GD at 10 than at 25 ºC, but there is no reasonable explanation for this result at present. The efficacy of CA^2WT DG for the decontamination of CARC panels at –15 ºC has been assessed with simulants and found to be more effective than simple rinsing alone.[14]

Table 22. Stirred-Reactor Data for CA^2WT DG at 25 and 10 ºC[a]

25 ºC									
Time	% HD			% VX			% GD		
10 min	16.6	12.6	7.8	22.2	32.6	35.1	4.6	2.7	9.1
20	0.7	9.3	0.6	12.2	15.9	21.7	0.1	0.3	1.2
30	0.0	1.0	0.1	3.7	7.7	12.7	0.0	0.0	0.0
40	0.1	0.1	0.3	1.7	4.2	6.1	-	-	-
50	0.0	0.1	0.3	0.6	1.8	2.4			
60	0.1	0.1	0.1	0.2	0.9	1.3			
10 ºC									
Time	% HD			% VX			% GD		
10	45.9	2.6	25.5	19.8	20.9	28.4	2.0	0.8	6.0
20	6.6	26.9	4.0	11.7	13.5	16.0	0.0	0.0	0.5
30	1.2	46.3	0.7	8.4	10.1	13.0	-	-	0.0
40	1.0	11.5	0.5	6.1	10.3	10.6			-
50	0.8	3.7	0.7	5.6	7.4	8.3			
60	0.6	1.2	0.6	4.1	5.3	6.0			

[a] 1 mL agent in 50 mL decon. Triplicate runs in stirred reactors. Results expressed as % agent remaining.

4. CONCLUSIONS

New Decon Green® remains a broad-spectrum decontaminant, effective against both chemical agents VX, GD, HD, and biological agents such as anthrax; possesses more than a 12 hr pot-life; and is suitable for use between 5 and 50 ºC. The reduced solvent content of New DECON GREEN® renders it less damaging to plastics and paint. However, it is not as effective as Decon Green® Classic in decontaminating CARC paint—for precisely the same reason: The reduced solvent content does not allow it to penetrate/soften CARC paint to react with sorbed agent. GD, VX, and especially HD soften CARC paint. CARC paint softened with GD, VX, and HD cannot be effectively decontaminated without the use of a penetrating, softening solvent. Although New Decon Green® is limited to use at temperatures above 5 ºC, the CA^2WT DG variant is suitable for use at temperatures down to –15 ºC. Compared to New Decon Green®, CA^2WT DG possesses similar efficacy on CARC surfaces at 25 ºC, and similar agent reactivity at 10 ºC.

The model presented to relate measured contact hazard levels to potential vapor hazard levels predicts levels far in excess of ORD vapor hazard levels when ORD contact hazard levels are present, as is invariably the case for sorptive surfaces such as CARC paint. Thus, the futility of bothering to measure such high vapor levels when they can be reasonably assured from contact hazard measurements. Moreover, current off-gassing measurement practices are quite subjective, leading to ambiguous results which cannot be straightforwardly-related to any particular vapor hazard level standard. Only contact hazard measurements and/or total extraction (residual hazard) remain the only unambiguous methods to verify decontamination efficacy on surfaces such as CARC, where substantial agent remains following decontamination.

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